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FORM PTO-1390 (REV 10-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEYS DOCKET NUMBER U. MINIERE-03
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/936531
INTERNATIONAL APPLICATION NO. PCT/EP00/00932	INTERNATIONAL FILING DATE 3 February 2000	PRIORITY DATE CLAIMED 9 February 1999	
TITLE OF INVENTION CENTRIFUGALLY ATOMIZED ZINC ALLOY POWDER FOR ALKALINE			BATTERIES.
APPLICANT(S) FOR DO/EO/US			

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

- ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
- ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
- ☒ This is an express request to promptly begin national examination procedures (35 U.S.C. 371(f)).
- ☐ The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).
- ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - ☒ is attached hereto (required only if not communicated by the International Bureau).
 - ☐ has been communicated by the International Bureau.
 - ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
- ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
- ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - ☐ are attached hereto (required only if not communicated by the International Bureau).
 - ☐ have been communicated by the International Bureau.
 - ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - ☐ have not been made and will not be made.
- ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
- ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 16 below concern document(s) or information included:

- ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
- ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
- ☐ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
- ☐ A substitute specification.
- ☐ A change of power of attorney and/or address letter.
- ☒ Other items or information:
Corrected Pages 8 and 9 of the Specification.

U.S. APPLICATION NO. 09/936531 INTERNATIONAL APPLICATION NO. _____ ATTORNEY'S DOCKET NUMBER _____							
17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1000.00 <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$860.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$710.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$690.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT =		CALCULATIONS PTO USE ONLY <table style="width:100%; border-collapse: collapse;"> <tr> <td style="width:50%; border-bottom: 1px solid black;">\$ 860.00</td> <td style="width:50%; border-bottom: 1px solid black;">860.00</td> </tr> <tr> <td style="border-bottom: 1px solid black;">\$ 130.00</td> <td style="border-bottom: 1px solid black;">130.00</td> </tr> </table>		\$ 860.00	860.00	\$ 130.00	130.00
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Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).		<table style="width:100%; border-collapse: collapse;"> <tr> <td style="width:50%; border-bottom: 1px solid black;">\$ 130.00</td> <td style="width:50%; border-bottom: 1px solid black;">130.00</td> </tr> </table>		\$ 130.00	130.00		
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CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE				
Total claims	10 - 20 =	0	X \$18.00				
Independent claims	3 - 3 =	0	X \$80.00				
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$270.00				
TOTAL OF ABOVE CALCULATIONS =			\$ 1,260.00				
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.							
SUBTOTAL =							
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).							
TOTAL NATIONAL FEE =			\$ 1,260.00				
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property							
TOTAL FEES ENCLOSED =			\$				
			Amount to be refunded: \$				
			charged: \$ 1,260.00				
a. <input type="checkbox"/> A check in the amount of \$ _____ to cover the above fees is enclosed. b. <input checked="" type="checkbox"/> Please charge my Deposit Account No. <u>50-1864</u> in the amount of \$ <u>1,260.00</u> to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>50-1864</u> . A duplicate copy of this sheet is enclosed.							
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.							
SEND ALL CORRESPONDENCE TO: MISHRIKAL JAIN NIRMAL & ASSOC. 11620 MASTERS RUN BELMONT CITY, MD, 21042 Tel: 410-995-0762							
SIGNATURE <u>Mishrikal Jain</u> MISHRIKAL JAIN NAME <u>29,315</u> REGISTRATION NUMBER							

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CENTRIFUGALLY ATOMIZED ZINC ALLOY POWDER FOR ALKALINE BATTERIES

This invention relates to centrifugal atomized zinc alloy powders for
5 alkaline batteries.

Zinc alloy powders are used as an ingredient of the anode active material
of alkaline batteries. Zinc alloy powders containing one or more elements
selected from In, Bi, Al, Ca and Pb, are known from US 5,082,622 and
10 WO 94/19502. Such alloys are normally prepared by traditional powder
manufacturing techniques, such as allowing the molten alloy to flow away in a
jet of compressed air. The use of a protective atmosphere in such gas jet
atomisation process is possible, but quite expensive because of the high flow
rate needed. Powders which are manufactured by compressed air atomisation
15 do not always have an acceptable resistance to corrosion in the electrolyte of
the battery, especially after partial discharge of the latter. A well-known way
of dealing with a low resistance to corrosion is by adding certain amounts of
organic or inorganic products. An example of an organic product is
polyethylene glycol. However this procedure also has some disadvantages,
20 e.g. the battery capacity could be decreased.

Centrifugal atomisation is a method in which molten metal is poured
from a nozzle on a high velocity rotating plate, a cup or a disk with a
predetermined shape whereby the metal is disintegrated by centrifugal force.
The above cited documents merely mention that the powders can also be
25 manufactured by centrifugal atomisation without specifying any working
condition. In these documents the particular effects of using centrifugal
atomized powders are not investigated.

According to the invention, a zinc alloy powder prepared by centrifugal
30 atomisation in a protective atmosphere with a reduced oxygen content has a
better resistance to corrosion than a powder prepared by atomisation in a
stream of air. Moreover, unacceptably low corrosion resistance values of some
of the above mentioned zinc alloy powders prepared by atomisation in air are
turned into acceptable values when the same alloys are prepared by
35 centrifugal atomisation in said protective atmosphere. The use of organic and
inorganic corrosion inhibitors can be limited, or even eliminated. The capacity
of batteries containing powders according to the invention is good. The

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production method is economically sound, because the consumption of gas in the protective atmosphere is very low.

The alloy powder of the invention consists of either

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(a) 0.005-2 %, and preferably 0.01-2 % by weight of indium; and 0.005-0.2 %, preferably 0.01-0.2 % by weight of either one of Al and Bi, or

(b) 0.005-2 % by weight of indium; and 0.005-0.2%, preferably 0.01-0.2 % by weight of Bi; and 0.001-0.5%, preferably 0.003-0.5 % of either one or both of Al

10 and Ca, or

(c) 0.005-2%, and preferably 0.01-2 % by weight of either one or both of Bi and Al,

and in each case 0-0.5 % by weight of Pb, the remainder being zinc.

15 By zinc is meant here and in the following thermally or electrolytically refined zinc (Special High Grade). In SHG zinc, the Pb content is normally limited to less than 30 ppm. For economical reasons the indium content can be limited to 5000 ppm, or even to 1000 ppm.

20 The powder is prepared by centrifugal atomisation, characterized in that this atomising process is performed in a protective atmosphere, mainly consisting of inert gasses, which controls the oxidation. More particularly, the oxygen content in the atmosphere is less than 4% by volume.

In order to prevent excessive oxidation, the oxygen content is limited to 4%, and preferably less than or equal to 3.5%. On the other hand, a minimum
25 amount of oxygen can be useful to control the shape of the solidified droplets. Also, too low oxygen contents imply a reduction of the capacity of the battery. For these reasons the oxygen content is preferably greater than 0%, and especially greater than or equal to 0.2%.

30 The rotating disk of the atomizer is made out of materials like alumina, carbon or fused silica, which can also be coated to prevent oxidation or occurrence of accretion.

Another object of the invention is a process to manufacture zinc alloy powders comprising the step of centrifugally atomising the zinc alloys mentioned above and characterized in that the centrifugal atomising process is
35 performed in a protective atmosphere, where the oxygen content is less than 4% by volume, and preferably greater than 0 % by volume. Most preferably the oxygen content is between 0.2 % and 3.5 % by volume.

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The applicant has found that when the powders described previously are prepared by a centrifugal atomising process in the above described protective atmosphere, they are consistently showing a better resistance to corrosion in the electrolyte of the battery, after partial discharge of the latter, compared to powders with identical compositions and prepared by air jet atomisation. The resistance to corrosion and the capacity of the powders according to the compositions of the invention, and prepared by centrifugal atomisation, are all recommendable for practical use in alkaline batteries. In a further experiment it is shown that the capacity of a battery using powders prepared by centrifugal atomisation according to the invention is better than when powders are used prepared by gas jet atomisation in a protected atmosphere with the same oxygen content.

In case the desired powder contains additives which can be cemented (for instance In), then another way to produce the powder consists in preparing a powder with the additives which cannot be cemented and optionally with a part of the additives which can be cemented according to the above mentioned method of centrifugal atomisation and making an anode from the thus obtained powder. That anode is introduced in the battery and the additives which can be cemented, are either added to the electrolyte of the battery, from which they cement on the powder of the anode, or they are cemented before the introduction in the battery. It is also possible to obtain powders for alkaline batteries by mixing a powder with a given composition and produced in a traditional way, such as atomisation in a stream of air, with a powder which was prepared by centrifugal atomisation in a protective atmosphere, according to the invention.

This invention relates not only to a powder such as it is introduced in the battery, but also to a powder such as it is present in the battery, and which consists entirely or partly of powder prepared by centrifugal atomisation, the remainder being prepared by traditional powder manufacturing techniques.

The powder is suitable for use in any type of battery, such as LR6, LR14, LR20.

Here, the following should be noted: Centrifugal atomized zinc alloy powders are mentioned in "*Atomization of melts for Powder Production and Spray Deposition*", by A.J. Yule and J. J. Dunkley ; Clarendon Press, Oxford; 1994; pp. 223-224. According to Yule, centrifugal atomisation is used as it gives a

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very closely controlled particle size distribution, which renders the powder production more economical. It is also possible, by control of the atmosphere inside the atomizer, to control the particle shape and hence the surface area and apparent density of the powder, both of which are significant for battery applications. This publication however does not mention which alloys can be exploited beneficially for this application, and neither is described what the controlled atmosphere inside the atomizer should be. Also it does not describe if and in what way the process of centrifugal atomisation has an influence on the characteristics of the batteries prepared with these powders.

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Experiment 1

In an example of producing the powder of the invention, the powders are prepared by adding the desired quantities of the alloying elements to molten zinc, which is homogenized by stirring at elevated temperature. The process of centrifugal atomisation is carried out as follows: about 150 kg of alloyed zinc is melted in a protective atmosphere at 480°C and poured on a rotating disk. The rotating disk is made of carbon, and placed 25 mm below the ejecting nozzle concentrically, 1.5 m above the bottom at the centre of the atomising chamber. The atomising chamber is filled with nitrogen and 0.4% to 3.3% of oxygen (see Table 1). The rotation speed is about 5000 rpm, the diameter of the disk is 170 mm and the flow rate of molten metal is about 530 kg/hr.

With the alloy powders batteries of the LR14-type are produced. These batteries are discharged over 2 Ohm for 1 hr or for 4 1/2 hrs. Subsequently one determines the quantity of hydrogen which is evolved when the batteries are maintained for 7 days at 71°C. The resulting gas production is expressed as ml of gas per battery. Also the volume expansion of a partially discharged anode is measured after 24 hrs at 71°C, and the volume increase is expressed as a percentage of the original volume. This measuring technique is an out-of-cell test described in US 5,364,715. Both in cell and out-of-cell gassings are a measure for the gassing after partial discharge.

The results of these tests are compared with results on traditional air atomized powders with the same chemical composition. In the examples, all powders are sieved on 500 micron to take away the very course particles. The results of Experiment 1 are summarized in Table 1.

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Table 1: experimental results of centrifugally atomised powders vs. powders atomised in a stream of air

Ex. No.	Alloy powder additives (ppm)				(*)	O ₂ % by Vol	Volume expansion (%)	Gassing after partial discharge 4 1/2 hrs, 2 Ohm (ml)	Gassing after partial discharge 1 hr, 2 Ohm (ml)
	In	Bi	Ca	Al					
(1)	150	230	-	-	C	0.4	3.4	4.63	1.95
(2)	500	100	-	100	C	0.4	1.7	2.07	1.5
(3)	200	180	-	70	C	0.4	3.6	3.67	1.7
(4)	250	250	150	-	C	0.4	3.1	3.57	1.37
(5)	-	250	-	-	C	0.4	6.5	8.05	3.12
(6)	2000	500	-	-	C	0.4	3.4	2.38	1.83
(7)	200	-	-	70	C	1.0	4.1	4.20	na
(8)	-	200	-	70	C	0.8	2.1	4.03	na
(9)	200	100	180	70	C	3.3	1.5	2.33	na
(10)	150	230	-	-	T	air	28.4	10.65	13.63
(11)	500	100	-	100	T	air	5.6	2.5	2.3
(12)	200	180	-	70	T	air	5.1	4	1.7
(13)	250	250	150	-	T	air	10.2	4.08	6.75
(14)	-	250	-	-	T	air	19.8	9.23	12.13
(15)	2000	500	-	-	T	air	41.9	12.7	19.58
(16)	200	-	-	70	T	air	43.6	19.53	na
(17)	-	200	-	70	T	air	4.5	6.13	na
(18)	200	100	180	70	T	air	2.4	4.07	na
(19)	500	5000	-	70	T	air	26.2	16.7	13.5
(20)	500	5000	-	70	C	0.4	30.1	13.5	6.37
(21)	600	-	-	-	T	air	87.6	52.63	(**)
(22)	600	-	-	-	C	0.4	42.1	25.07	22.43
(23)	900	-	-	-	C	0.4	25.9	21.38	15.9
(24)	40	40	-	-	C	0.4	28.5	11.63	8.57

5 (*): 'C': centrifugal atomized powder; 'T': powder preparation by traditional air jet atomisation

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(**): Leakage occurred, which indicates that gassing was excessive and unacceptable.

na: not available

- 5 For the volume expansion, a value of less than 12% is considered to be very good, preferably the expansion should be less than 8%.

The gassing after partial discharge is good for values less than 12 ml, and preferably less than 8 ml, for both the gassing tests after 4½ hrs or 1 hr of discharge.

- 10 Comparison of Examples No. (1)-(9) with Examples No. (10)-(18) shows that the powders according to the invention have better corrosion resistance and expansion parameters than traditionally prepared powders. The compositions of the Examples No. (10), (14), (15) and (16), which do not yield acceptable results when prepared in the traditional way, have excellent results
- 15 when prepared according to the invention, as shown in Examples No. (1), (5), (6) and (7). Examples No. (20), (22)-(24) show that centrifugal atomisation is not yielding satisfactory results for zinc powders consisting of alloys other than the compositions claimed in the invention.

- 20 It has been noticed that the powder capacity of the powders prepared by centrifugal atomisation in this experiment and used in LR-6 batteries is varying between 90 % and 110 % as compared to a standard powder used in these batteries nowadays, for instance the powder according to Example No. (12) above.

25 Experiment 2

- In Experiment 2 a comparison is made between powders which were made exclusively by centrifugal atomisation but with different oxygen contents in the protective atmosphere. The other process parameters and the
- 30 test procedure of the LR-14 batteries using the powders are identical to Experiment 1. The results are presented in Table 2.

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Table 2: Volume expansion of different centrifugally atomised powders

Ex. No.	Alloy powder additives (ppm)					O ₂ % by Volume	Volume expansion (%)
	In	Bi	Ca	Al	Pb		
(25)	200	100	-	100	54	2.5	3.0
(26)	150	150	-	-	40	2.0	5.6
(27)	200	100	-	100	54	5	12.3
(28)	150	150	-	-	40	air	23.6

5 The results of this experiment are the following:

- when the oxygen content in the protective atmosphere of the centrifugal atomising chamber is outside the limits of this invention, then the volume expansion measured becomes unacceptable, as is shown when comparing Examples No. (25) and (26), with (27) and (28).

10

Experiment 3

15 In Experiment 3 powders prepared by traditional gas jet atomisation under a protective atmosphere are compared with powders prepared by centrifugal atomisation under the same protective atmosphere. The procedure for the centrifugal atomisation is identical to the procedure in Experiment 1. The powders were tested in LR-6 batteries, which were discharged at a constant current of 1000 mA. A standard air atomised powder used in LR-6

20 batteries nowadays, i.e. the powder Example No. (12) from Table 1, was used as a reference. The discharge time to a residual voltage of 1.0 V for the different powders is expressed as a percentage of the discharge time to 1.0 V for the reference powder. The discharge time is a measure which expresses the capacity of the tested batteries. The results are given in Table 3 below.

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Table 3: Discharge time of batteries prepared with centrifugally atomised vs. gas jet atomised powders

Ex. No.	Alloy powder additives (ppm)					(***)	O ₂ % by Volume	Discharge time (%)
	In	Bi	Ca	Al	Pb			
(29)	160	230	-	-	46	C	2.35	102
(30)	160	230	-	-	46	C	0.6	90
(31)	160	230	-	-	46	G	2.35	87
(32)	160	230	-	-	46	G	0.6	61

(**): 'C': centrifugal atomized powder; 'G': powder preparation by gas jet

5 atomisation

The results of Experiment 3 are the following:

- both discharge times for centrifugally atomised powders are within the claimed range of 90-110% of the discharge time of the standard powder. 2.35%

10 O₂ yields a better capacity than 0.6% O₂.

- both discharge times for the gas atomised powder are significantly lower than the discharge times of powders atomised centrifugally in gas with the same composition of the powder and of the protective atmosphere, and are less than 90%.

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In Table 5 additional results for powders prepared by centrifugal atomisation according to the invention are given. They have been prepared and tested following the procedure of Experiment 1.

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Table 5: Volume expansion of different centrifugally atomised powders

Ex. No.	Alloy powder additives (ppm)					O ₂ % by Volume	Volume expansion (%)
	In	Bi	Ca	Al	Pb		
(33)	250	250	150	-	-	0.6	3.3
(34)	500	110	-	35	-	0.6	1.9
(35)	500	110	-	35	-	1.35	2.5
(36)	500	110	-	35	-	1.6	2.1
(37)	500	110	-	35	-	2.1	6.7
(38)	500	110	-	35	-	2.6	7.6
(39)	150	230	-	-	-	1.6	5.5
(40)	200	100	-	100	-	1.6	4.0
(41)	200	100	-	100	-	2.6	2.9
(42)	150	230	-	-	-	0	5.6
(43)	150	230	-	-	-	2	7.3
(44)	200	100	-	100	40	2	2.7
(45)	150	230	-	-	38	2.45	6.7
(46)	150	230	-	-	55	0.25	5.0
(47)	200	100	-	100	40	3.5	3.7

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CLAIMS

1. A centrifugal atomized zinc alloy powder for alkaline batteries consisting of either of
- 5 (a) 0.005-2 % by weight of indium, and 0.005-0.2 % by weight of either one of Al and Bi, or
- (b) 0.005-2 % by weight of indium, and 0.005-0.2 % by weight of Bi, and 0.001-0.5 % of either one or both of Al and Ca, or
- (c) 0.005-2 % by weight of either one or both of Bi and Al,
- 10 and 0-0.5 % by weight of Pb, the remainder being zinc, and characterized in that the centrifugal atomising process is performed in a protective atmosphere, where the oxygen content is less than 4% by volume.
2. A centrifugal atomized zinc alloy powder according to claim 1, consisting of
- 15 either of
- (a) 0.01-2 % by weight of indium, and 0.01-0.2 % by weight of either one of Al and Bi, or
- (b) 0.005-2 % by weight of indium, and 0.01-0.2 % by weight of Bi, and 0.003-0.5 % of either one or both of Al and Ca, or
- 20 (c) 0.01-2 % by weight of either one or both of Bi and Al, and 0-0.5 % by weight of Pb, the remainder being zinc.
3. A centrifugal atomized zinc alloy powder in alkaline batteries according to claim 1 or 2, characterized in that the oxygen content in the protective atmosphere is greater than 0 % by volume.
- 25 4. A centrifugal atomized zinc alloy powder in alkaline batteries according to claim 3, characterized in that the oxygen content in the protective atmosphere is between 0.2 % and 3.5% by volume.
5. An alkaline battery consisting of an anode, a cathode and an electrolyte, characterized in that the battery uses a centrifugal atomized zinc alloy
- 30 powder according to one of the claims 1-4.
6. An alkaline battery according to claim 5, characterized in that the powder comprises metal cemented out of the electrolyte.
7. A process for the manufacturing of a zinc alloy powder for alkaline batteries, comprising the step of centrifugally atomising a zinc alloy
- 35 consisting either of
- (a) 0.005-2 % by weight of indium, and 0.005-0.2 % by weight of either one of Al and Bi, or

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- (b) 0.005-2 % by weight of indium, and 0.005-0.2 % by weight of Bi, and 0.001-0.5 % of either one or both of Al and Ca, or
- (c) 0.005-2 % by weight of either one or both of Bi and Al, and 0-0.5 % by weight of Pb, the remainder being zinc, characterized in
- 5 that the centrifugal atomising process is performed in a protective atmosphere, where the oxygen content is less than 4% by volume.
8. A process for the manufacturing of a zinc alloy powder for alkaline batteries, comprising the step of centrifugally atomising a zinc alloy consisting either of
- 10 (a) 0.01-2 % by weight of indium, and 0.01-0.2 % by weight of either one of Al and Bi, or
- (b) 0.005-2 % by weight of indium, and 0.01-0.2 % by weight of Bi, and 0.003-0.5 % of either one or both of Al and Ca, or
- (c) 0.01-2 % by weight of either one or both of Bi and Al,
- 15 and 0-0.5 % by weight of Pb, the remainder being zinc, characterized in that the centrifugal atomising process is performed in a protective atmosphere, where the oxygen content is less than 4% by volume.
9. A process according to claims 7 or 8, characterized in that the oxygen content in the protective atmosphere is greater than 0 % by volume.
- 20 10. A process according to claims 7 to 9, characterized in that the oxygen content in the protective atmosphere is between 0.2 % and 3.5% by volume.

SECRET-05104

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION☒ Original☐ Supplemental☐ Substitute☐ PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below), or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

CENTRIFUGALLY ATOMIZED ZINC ALLOY POWDER FOR ALKALINE BATTERIES

(Title of the Invention)

the specification of which (check one)

☐ is attached hereto☒ was filed on 3 February 2000 as U. S. Application Serial Number or PCT

International Application Number PCT/EP00/00932

and was amended _____

(if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a) - (d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified, by checking the box below, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Applications			Priority Claimed		Copy Attached	
Application Number	Country	Foreign Filing Date (MM/DD/YYYY)	YES	NO	YES	NO
99200353.3	EPO	09/02/1999	X		X	

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below and claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

Inventors: _____

For: _____

Filed: _____

Declaration for Patent Application

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Attorney Docket No.: U.MINIERE-03

Parent Application Number	Filing Date	Status (Mark Appropriate Column Below)		
		Patented	Pending	Abandoned

As a named inventor, I hereby revoke all prior powers and appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

FIRM NAME: NIRMEL & ASSOCIATES, 1730 K Street, N.W., Suite 304, Washington, DC 20006. USA.

ATTORNEY AND/OR AGENT	REGISTRATION NO.
MISHRILAL JAIN	<u>29,315</u>
CHITTARANJAN NIRMEL	<u>30,408</u>

ATTORNEY AND/OR AGENT	REGISTRATION NO.

I acknowledge the above-listed attorneys and agents and their firm Nirmel & Associates, represent my employer (if I am an employee and this application has been or will be assigned to my employer) or the entity with which I have contracted (if I am an independent contractor and this application has been or will be assigned to such entity) and in such cases do not represent me individually. I further acknowledge I have not established, nor will I seek to establish, any personal attorney/client relationship with Nirmel & Associates in connection with this application and understand that, should I require legal representation, I will obtain such, at my expense, other than through Nirmel & Associates.

Send Correspondence to: Mishrilal Jain, Esq.
11620 Masters Run
Ellicott City, MD. 21042-1537

Direct telephone calls to: Dr. Mishrilal Jain (410) 995-0762

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor STRAUVEN, Yvan

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For: _____

Filed: _____

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Full name of second inventor GAY, Bruno

Inventor's signature Bruno Gay Date 23-08-01

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Full name of third inventor N/A

Inventor's signature _____ Date _____

Residence _____

Citizenship _____

Post Office Address _____